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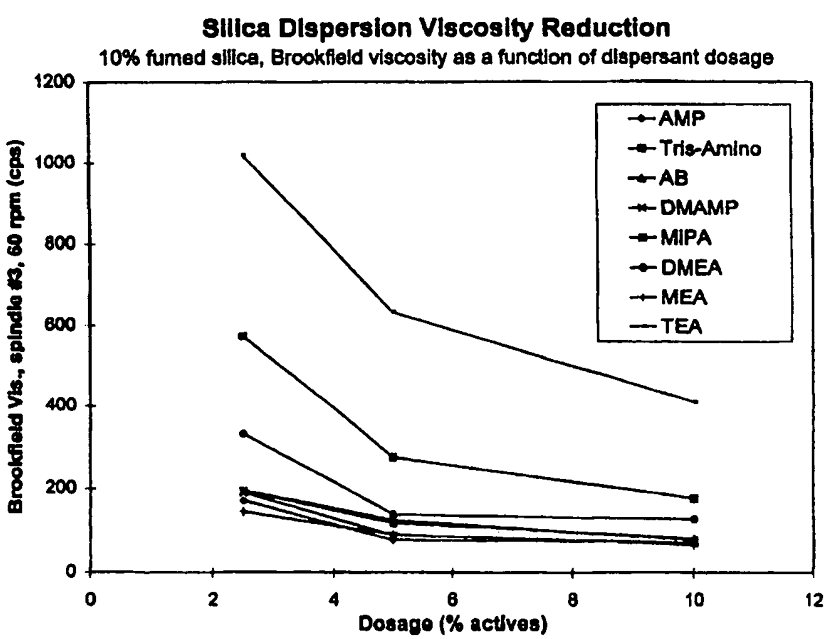


(57) Abstract

Stable aqueous silica dispersions are produced by adding effective amounts of certain primary, secondary, and tertiary amino alcohols as defined herein. Such dispersions remain non-dilatant, even under high stress.

STABILIZATION OF SILICA DISPERSIONS

BACKGROUND OF THE INVENTION

This invention relates generally to aqueous dispersions of fine particles of silica. Such dispersions have many uses, including manufacture of ceramics, coatings, polishes, dentifrices, etc. Silica dispersions characteristically thicken, that is, they are said to be "dilatant" when subjected to shearing conditions, such as may occur in manufacturing processes. Consequently, it is important in many applications that the viscosity of the dispersion be reduced, and thickening or gelling be prevented.

Viscosity is commonly measured with the Brookfield instrument, in which a spindle is rotated at a given speed in the fluid being measured and the resistance correlated to viscosity. While many common liquids are classified as Newtonian, that is, they have a constant viscosity regardless of the shearing conditions applied, silica dispersions tend to become less viscous at first and then to become dilatant. Their viscosity appears to become lower as the shear stress is increased but, at some shear level, they gel and the viscosity increases substantially. This phenomenon is undesirable since gelling can cause problems in handling silica dispersions.

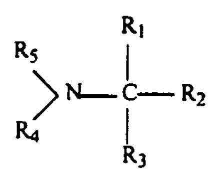
There are a number of patents which concern methods of producing highly concentrated silica dispersions which are said to be stable. Many relate to the polishing of silicon wafers in the electronics industry. A recent patent, U.S. Patent No. 5,246,624, discloses a process for producing a stable dispersion containing more than 40% silica by first adding fumed silica to acidified water and then adding more water and raising the pH to between 7.0 and 12.0 by adding a stabilizer. The patent examples show the use of HCl to acidify the water and NH_4OH as a base to raise the pH and stabilize the dispersion. In U.S. Patent No. 4,892,612, the patentee discloses as his invention a method of polishing silicon wafers using a silica sol which contains larger than usual amounts of alkanolamines. The use of alkanolamines (also called herein "amino alcohols") is discussed also in "Practical Dispersion: A Guide to Understanding and Formulating Slurries," R. Conley, VCH Publishers, Inc., 1996, pp. 141-2. In one graph, the effect of the dosage of 2-amino-2-methyl-1-propanol (AMP) on the Brookfield viscosity is shown. For silica, in the range of 0-3% AMP, the viscosity of an aqueous dispersion of silica was shown to steeply decline as more AMP was added.

In general, the art considers pH an indicator of the stability of a silica dispersion. A pH above 7 results when various bases are added to stabilize silica dispersions. Both inorganic and organic bases have been used. Of the organic bases, amines and amino alcohols are known. The art, however, seems to have failed to show how to select amines, or that there are significant differences among them.

The present inventor has examined the effect of amines and found that there are differences in their ability to maintain stable silica dispersions under high shear conditions, as will be discussed and demonstrated below.

SUMMARY OF THE INVENTION

The invention relates generally to the formation of stable aqueous silica dispersions. In particular, it relates to silica dispersions which are not dilatant, that is, they do not gel even when subjected to high shear stress above about 10 Pa. It has been found that certain amino alcohols are capable of providing such dispersions. Preferred amino alcohols are those selected from the group consisting of primary, secondary, and tertiary amines in which the nitrogen atom of the amine is bonded to a secondary or tertiary carbon atom. Particularly preferred are amino alcohols defined by the following formula:



where R_1, R_2 and R_3 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl with the proviso that no more than one of R_1, R_2 and R_3 is H; and R_4 and R_5 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl.

In one aspect, the invention is a stable silica dispersion in which an effective amount of the amino alcohols is used, in particular, in an amount between about 1-10 wt %, based on the amount of silica solids present. Especially preferred amino alcohols are 2-amino-2-methyl-1-propanol (AMP), 2-amino-2-ethyl-1,3-propanediol (AEPD), 2-amino-2-methyl-1,3-propanediol (AMPD), 2-amino-2-hydroxymethyl-1,3-propanediol (TA), and 2-amino-1-butanol (AB).

The amount of silica present in dispersions according to the invention typically is about 1-30 wt % based on the water present, although greater or lesser amounts are believed to be possible. The silica particles generally will have an effective diameter up to about 10 μm, typically 1.5 μm or less, which is within the range typically considered to provide colloidal suspensions.

In another aspect, the invention is a method of producing stable aqueous silica dispersions. Predetermined amounts of one or more of the selected amino alcohols are added to the amount of water which is required to provide the desired silica concentration. Then, the predetermined amount of silica particles is added with mixing to produce the stable silica dispersions. The amino alcohols are those defined above. The silica dispersion typically will contain about 1-30 wt % silica particles having an effective diameter up to 10 μm, typically 1.5 μm or less and about 1-10 wt % of the amino alcohols based on the amount of silica solids. Other additives, such as biocides, pH modifiers, and dispersants, may be included.

In still another aspect of the invention, a stable silica dispersion is produced which remains non-dilatant even under high shear stress conditions above about 10 Pa.

BRIEF DESCRIPTION OF THE DRAWINGS

Other objects and advantages of the invention will become apparent upon reading the following detailed description and upon reference to the drawings.

Figure 1 is a graph of the effect of varying the concentration of various bases on the measured Brookfield viscosity.

Figure 2 is a bar chart comparing the stability of silica dispersions stabilized by various bases.

Figures 3A-O are graphs showing the effect of increasing shear stress on viscosity for various bases.

Figure 4 is a graph showing the maximum obtainable amount of silica solids without gellation of the dispersion.

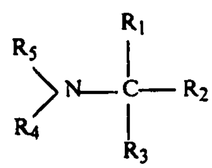
DESCRIPTION OF ILLUSTRATIVE EMBODIMENTS

Silica Particles

Fine silica particles are used in many applications. Such particles may be made by several known methods, such as the vapor phase hydrolysis of chlorosilanes to produce fumed silica. The particles may vary in size up to about 10 μm, typically they will be about 0.01-1.5 μm, in the range of colloidal suspensions. For purposes of this invention, the size of the particles is defined by their average effective diameter, the effective diameter being a known means for defining the size of irregular particles. Alternatively, some silica particles are substantially spherical and such particles may also be used in the method of the invention. It is not believed that the source of the silica particles is an important factor in the success of the method of the invention.

Amino Alcohols

The amino alcohols of the invention are primary, secondary, or tertiary amines in which the nitrogen atom is bonded to a secondary or tertiary carbon atom. Preferably, they are defined by the following formula:



where R_1, R_2 and R_3 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl with the proviso that no more than one of R_1, R_2 and R_3 is H; and R_4 and R_5 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl.

It will be shown in the Examples below that the amino alcohols just defined are superior to other amines and inorganic bases. In particular, they are able to better prevent gelling of the silica dispersion under high shear stress conditions, that is, they maintain the silica suspension in a non-dilatant state where other bases fail to do so. Also, they improve the shelf life stability of the dispersions, that is, they maintain the fluidity of the dispersions during storage for longer periods than other bases.

Especially preferred are 2-amino-2-methyl-propanol (AMP), 2-amino-2-ethyl-1,3-propanediol (AEPD), 2-amino-2-methyl-1,3-propanediol (AMPD), 2-amino-2-

hydroxymethyl-1,3-propanediol (TA), and 2-amino-1-butanol (AB). They may be used alone or in mixtures.

The reason for the unexpected superiority of these amino alcohols is not entirely clear, but at present it is believed that the presence of the amine group on the secondary or tertiary carbon of the alkanol, rather than on the primary carbon atom characteristic of many other amino alcohols, contributes to the surprising effectiveness of the amines of the invention.

Silica Dispersions

Silica dispersions have generally been made using a base in order to raise the pH and to stabilize a silica-water mixture. Other materials may be added to improve the stability and useful life of the dispersion, such as biocides, pH modifiers, dispersants and the like known in the art. From the U.S. patents noted above and elsewhere, one skilled in the art could conclude that either inorganic bases, such as KOH and NH_4OH, or organic bases, such as amines and amino alcohols, may be roughly equivalent in their effect on silica dispersions. The present inventor has found that that conclusion is not correct, at least as far as the primary, secondary, and tertiary amino alcohols of the invention are concerned. As will be shown, the choice of base does affect the pH of the dispersion. The pH does not, however, appear to be an indicator of the effectiveness of the base for purposes of this invention. The amino alcohols of the invention provide dispersions with pH values generally lower than are provided by equal dosages of KOH or NH_4OH.

The present inventor has examined the effect on silica dispersions of many bases, both inorganic and organic. In particular, the inventor has determined the effect of a range of shear stress values on the viscosity of silica dispersions. He has found that silica dispersions show a decreasing viscosity with increasing amine concentration as measured by the Brookfield instrument, which is consistent with the disclosed performance in the Conley text mentioned above. The inventor has also found, however, that with some bases, gelling occurs when the shear stress is further increased, that is, the silica dispersion becomes dilatant. When the amino alcohols of the invention are used to stabilize the silica dispersion, they are non-dilatant under stress conditions where other bases are unable to maintain a low viscosity and gelling occurs. Consequently, the

amino alcohols of the invention provide the user with an improved silica suspension which remains fluid under high stress conditions and avoids gelling. It is believed that stress conditions of the Examples are typical of those encountered by silica dispersions in many of their applications. Consequently, it is important that they do not gel while being handled or used. Examples of shear stresses experienced in specific applications are shown in Table 1 (H. A. Barnes, J. F. Hutton and K. Walters, *An Introduction To Rheology*, Elesiever, Amsterdam, 1989, p. 13).

TABLE 1

Situation	Typical Range Of Shear Rates (s^{-1})	Range Of Shear Stresses (Pa), for 0.1-1.0 Pa.s Viscosity	Application
Extrusion	1-100	0.1-100	Polymer Manufacturing
Chewing. Swallowing	10-100	1-100	Foods
Dip Coating	10-100	1-100	Paints
Mixing. Stirring	10-1000	1-1000	Manufacturing Liquids
Pipe Flow	1-1000	0.1-1000	Pumping
Spraying, Brushing	1000-10,000	100-10.000	Spray-drying, Painting
Rubbing	10,000-100,000	1000-100,000	Application Of Lotion On Skin
Milling Pigments In Fluids	1000-100.000	100-100,000	Paints, Inks
High Speed Coating	100,000-1,000,000	10.000-1.000.000	Paper
Lubrication	1000-10.000.000	100-10.000.000	Gas Engines

While the concentration of dispersed silica particles is not especially limited so far as is currently known, the silica dispersions of the invention typically are those containing about 1-30 wt % silica based on the water present. Larger or smaller concentrations are believed to be feasible. It is believed that higher or lower silica concentrations would also be improved by use of the amino alcohols of the invention.

Silica dispersions may be made by first mixing the desired amount of the selected amino alcohol(s) to the amount of water required to yield the desired final concentration of silica in water. Typically, the amount of the amino alcohol(s) used will be in the range of about 1-10 wt % based on the amount of silica solids present. Thereafter. the desired amount of silica particles will be added and mixed with the amine and water to produce the final silica dispersion.

EXAMPLE 1

Dispersions of 10% (by weight) M5 Cab-O-Sil Fumed Silica were prepared by mixing the silica with water containing 2.5%, 5%, or 10% (by weight based on silica solids) of either 2-amino-2-methyl-1-propanol (AMP), 2-amino-2-hydroxymethyl-1,3-propanediol (Tris-Amino or TA), 2-amino-1-butanol (AB), 2-dimethylamino-2-methyl-1-propanol (DMAMP), monoisopropanolamine (MiPA), dimethylethanolamine (DMEA), monoethanolamine (MEA), or triethanolamine (TEA). The relative viscosity of each sample was then measured using a Brookfield viscometer, using spindle #3 at 60 rpm.

The results are shown in Figure 1, which shows that monoethanolamine (MEA), 2-amino-2-methyl-1-propanol (AMP), and 2-dimethylamino-2-methyl-1-propanol (DMAMP) reduce the silica dispersions viscosity better than any of the other five amines tested. Based on these results, one could conclude that monoethanolamine (MEA), 2-amino-2-methyl-1-propanol (AMP), and 2-dimethylamino-2-methyl-1-propanol (DMAMP) are the best silica dispersion stabilizers of those amines tested.

EXAMPLE 2

Dispersions of 10% (by weight) M5 Cab-O-Sil Fumed Silica were prepared by mixing the silica with water containing 2.5% (by weight based on silica solids) of either KOH, ammonia, monoethanolamine (MEA), monoisopropanolamine (MiPA), 2-amino-2-methyl-1-propanol (AMP), 2-amino-1-butanol (AB), 2-dimethylamino-2-methyl-1,3-propanediol (DMAMPD), 2-dimethylamino-1-butanol (DMAB), 2-dimethylamino-2-methyl-1-propanol (DMAMP), diethanolamine (DEA), 2-amino-2-ethyl-1,3-propanediol (AEPD), 2-amino-2-hydroxymethyl-1,3-propanediol (Tris-Amino or TA), 2-dimethylamino-2-ethyl-1,3-propanediol (DMAEPD), 2-amino-2-methyl-1,3-propanediol (AMPD), or triethanolamine (TEA). The pH of each sample was then measured.

The results in Table 2 show that KOH, ammonia, monoethanolamine, and monoisopropanolamine were the best pH modifiers of the fifteen chemicals tested. Based on these results, one could conclude that KOH, ammonia, monoethanolamine (MEA), and monoisopropanolamine (MiPA) are the best silica dispersion stabilizers of those amines tested.

TABLE 2

Base	pH
KOH	10.468
Ammonia	10.038
MEA	9.806
MiPA	9.532
AMP	9.357
L-AB	9.278
DMAMPD	9.214
DMAB	9.195
DMAMP-80	8.981
DEA	8.884
AEPD	8.85
TA	8.716
DMAEPD	8.537
AMPD	8.445
TEA	8.303

EXAMPLE 3

Dispersions of 10% (by weight) M5 Cab-O-Sil Fumed Silica were prepared by mixing the silica with water containing 2.5% (by weight based on silica solids) of either 2-amino-2-methyl-1-propanol (AMP), 2-dimethylamino-2-methyl-1-propanol (DMAMP), monoethanolamine (MEA), triethanolamine (TEA), KOH, or ammonia. The following qualitative test for the stabilizing effects of the amines was then performed.

The dispersion samples were stored in jars, undisturbed, on the shelf. Once daily, each jar was shaken rapidly up and down twice by hand, and the dispersions were examined to determine whether gellation had occurred.

The results are shown in Figure 2. 2-amino-2-methyl-1-propanol (AMP) and 2-dimethylamino-2-methyl-1-propanol (DMAMP) both stabilized the silica dispersions for significantly longer than any of the other four chemicals tested.

The results of Examples 1-3 show qualitatively that neither viscosity reduction nor pH are an accurate measure of overall silica dispersion stability, and that stability may depend on the selection of the base used as a stabilizer.

EXAMPLE 4

Dispersions of 10% (by weight) M5 Cab-O-Sil Fumed Silica were prepared by mixing the silica with water containing 1.25%, 2.5%, and 5.0% (by weight based on silica solids) of either 2-amino-2-methyl-1-propanol (AMP), monoethanolamine (MEA), KOH, ammonia, monoisopropanolamine (MiPA), 2-amino-1-butanol (AB), 2-dimethylamino-2-methyl-1,3-propanediol (DMAMPD), 2-dimethylamino-1-butanol (DMAB), 2-dimethylamino-2-methyl-1-propanol (DMAMP), diethanolamine (DEA), 2-amino-2-ethyl-1,3-propanediol (AEPD), 2-amino-2-hydroxymethyl-1,3-propanediol (Tris-Amino or TA), 2-dimethylamino-2-ethyl-1,3-propanediol (DMAEPD), 2-amino-2-methyl-1,3-propanediol (AMPD), or triethanolamine (TEA). The following quantitative test for the stabilizing effects of the amines was then performed.

To characterize the stability of the dispersions over a range of shear stresses, viscosity as a function of shear stress was measured using a research-grade, controlled-stress air-bearing rheometer (TA Instruments AR1000). A cone-and-plate geometry was used, wherein a small quantity of dispersion sample was poured onto a plate, and then a cone was lowered onto the sample. The cone used had an angle of 1:59:00 (degrees:minutes:seconds), and was truncated at the tip by 63 μm, so that the tip of the cone did not touch the plate, where it would interfere with the viscosity measurement. The dispersion sample filled the gap between the cone and the plate. The cone was then rotated at a controlled shear stress, and the rotational speed (or shear rate) was measured automatically by the rheometer by an optical coder. Viscosity was then calculated, and recorded as a function of shear stress.

For the purposes of this invention, a dispersion is deemed stable in high shear conditions by the results of the test described here. If the dispersion sample does not show severe shear-thickening effects, or gel, at shear stresses above about 20 Pa, then that dispersion is deemed to be stable in conditions of high shear.

In Figures 3A-3O, viscosity is graphed as a function of shear stress. When the slope of a curve in such a graph is negative, the dispersion is shear-thinning. When the slope of a curve in such a graph is positive, the dispersion is shear-thickening or dilatant. When the viscosity of a dispersion increases at least five-fold as the shear stress is merely

tripled (or less), the dispersion is said to have been severely dilatant, or to have gelled. In some instances, the samples appear to be severely shear-thickening at shear stresses above 10 Pa and then shear-thinning at shear stresses nearing 100 Pa, *e.g.*, 2.5% DMAMP in Figure 3F. This is believed to be an artifact of the measurement method. When a sample gels and changes from a fluid state to a gel state, the loss of the fluidity results in a loss of contact between the rotating cone of the rheometer at high rotational speeds. Thus, the cone rotates more easily and the measured viscosity appears to go down.

Notice in Figure 3A that each of the three dispersions tested over the dosage range, using 2-amino-2-methyl-1-propanol (AMP), were shear-thinning over the range of shear stresses tested. None of the samples showed any shear-thickening effects, nor did any of the samples gel during the course of the measurement.

Notice in Figure 3B that each of the three dispersions tested over the dosage range, using 2-amino-2-ethyl-1,3-propanediol (AEPD), were shear-thinning over the range of shear stresses tested. None of the samples showed any shear-thickening effects, nor did any of the samples gel in the rheometer during the course of the measurement.

Notice in Figure 3C that each of the three dispersions tested over the dosage range, using 2-amino-2-methyl-1,3-propanediol (AMPD), were shear-thinning over the range of shear stresses tested. None of the samples showed any shear-thickening effects, nor did any of the samples gel in the rheometer during the course of the measurement.

Notice in Figure 3D that each of the three dispersions tested over the dosage range, using 2-amino-2-hydroxymethyl-1,3-propanediol (TA), were shear-thinning over the range of shear stresses tested. None of the samples showed any shear-thickening effects, nor did any of the samples gel in the rheometer during the course of the measurement.

Notice in Figure 3E that each of the three dispersions tested over the dosage range, using 2-amino-1-butanol (AB), were shear-thinning over the range of shear stresses tested. None of the samples showed any shear-thickening effects, nor did any of the samples gel in the rheometer during the course of the measurements.

Notice in Figure 3F that the dispersion tested dosed at 5.0%, using 2-dimethylamino-2-methyl-1,3-propanediol (DMAMPD), was shear-thinning over the

range of shear stresses tested. The dispersions dosed at 1.25% and 2.5% showed shear-thickening effects at shear stresses above 25 Pascals. None of the samples gelled in the rheometer during the course of the measurements.

Notice in Figure 3G that the dispersion tested dosed at 5.0%, using 2-dimethylamino-1-butanol (DMAB), was shear-thinning over the range of shear stresses tested. The dispersions dosed at 1.25% and 2.5% showed shear-thickening effects at shear stresses above 25 Pascals. None of the samples gelled in the rheometer during the course of the measurements.

Notice in Figure 3H that the dispersion tested dosed at 5.0%, using 2-dimethylamino-2-methyl-1-propanol (DMAMP), was shear-thinning over the range of shear stresses tested. The dispersions dosed at 1.25% and 2.5% showed shear-thickening effects at shear stresses above 25 Pascals. None of the samples gelled in the rheometer during the course of the measurements.

Notice in Figure 3I that the dispersion tested dosed at 5.0%, using 2-dimethylamino-2-ethyl-1,3-propanediol (DMAEPD), was shear-thinning over the range of shear stresses tested. The dispersions dosed at 1.25% and 2.5% showed shear-thickening effects at shear stresses above 25 Pascals. None of the samples gelled in the rheometer during the course of the measurements.

Notice in Figure 3J that each of the three dispersions tested over the dosage range, using ammonia, were shear-thickening at shear stresses above 7 Pa. In fact, each sample gelled in the rheometer during the course of the measurements.

Notice in Figure 3K that each of the three dispersions tested over the dosage range, using monoethanolamine, were severely shear-thickening at shear stresses above 10 Pa. In fact, the sample treated with 1.25% MEA was too viscous to make a recordable measurement at stresses below 10 Pa and each sample gelled in the rheometer during the course of the measurements.

Notice in Figure 3L that each of the three dispersions tested over the dosage range, using diethanolamine (DEA), were shear-thickening at shear stresses above 10 Pa. In fact, the sample treated with 1.25% DEA was too viscous to make to recordable measurement at stresses below 100 Pa and each gelled in the rheometer during the course of the measurements.

Notice in Figure 3M that each of the three dispersions tested over the dosage range, using triethanolamine (TEA), were shear-thickening at shear stresses above 20 Pa. In fact, each sample gelled in the rheometer during the course of the measurements.

Notice in Figure 3N that each of the three dispersions tested over the dosage range, using monoisopropanolamine (MiPA), were shear-thickening at shear stresses above 10 Pa. In fact, each sample gelled in the rheometer during the course of the measurements.

Notice in Figure 3O that each of the three dispersions tested over the dosage range, using KOH, were shear-thickening at shear stresses above 20 Pa. In fact, the samples were too viscous to make a recordable measurement at stresses below 10 Pa, and each sample gelled in the rheometer during the course of the measurements.

These results show quantitatively that prior art teaches incorrectly that viscosity and pH modification is an accurate measure of overall silica dispersion stability. These results also clearly show that amino alcohols of a certain structure stabilize silica dispersions, and that amino alcohols without such structure do not.

EXAMPLE 5

Dispersions of 1%, 2%, 4%, 8%, 10%, 16%, and 26% (by weight) M5 Cab-O-Sil Fumed Silica were prepared by mixing the silica with water containing 5% (by weight based on silica solids) of either 2-amino-2-methyl-1-propanol (AMP), 2-dimethylamino-2-methyl-1-propanol (DMAMP), KOH, or ammonia. These dispersions were prepared to determine the maximum silica solid level obtainable by using such additives listed as a stabilizer. The maximum silica concentration was determined as the highest percent silica dispersion that could be prepared without gellation occurring during sample preparation.

Notice in Figure 4 that the use of an amino alcohol, as opposed to ammonia or KOH, enables the preparation of stable silica dispersions with higher solid levels.

It is concluded that the amino alcohols described in the present invention effectively disperse silica in such a way as to enable preparation of higher solid dispersions than can be prepared using the standard pH modifiers and viscosity modifiers.

While the present invention has been described with reference to one or more particular embodiments, those skilled in the art will recognize that many changes may be made thereto without departing from the spirit and scope of the present invention. Each of these embodiments and obvious variations thereof is contemplated as falling within the spirit and scope of the claimed invention, which is set forth in the following claims.

WHAT IS CLAIMED IS:

1. A stable aqueous silica dispersion comprising:

(a) silica particles having an average diameter up to about 10 μm;

(b) sufficient water to provide a predetermined concentration of said silica particles;

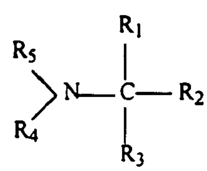
(c) an amount effective to stabilize the dispersion said silica particles in water of at least one amino alcohol from the group consisting of primary, secondary, and tertiary amines in which the nitrogen atom of the amine is bonded to a secondary or tertiary carbon atom.

2. A silica dispersion of Claim 1, wherein the amount of silica of (a) is about 1-30 wt % based on the water of (b).

3. A silica dispersion of Claim 1, wherein the effective amount of the amino alcohol of (c) is about 1-10 wt % based on the silica solids of (b).

4. A silica dispersion of Claim 1, wherein the silica particles have an average diameter of about 0.01 to 1.5 μm.

5. A silica dispersion of Claim 1, wherein said amino alcohols are defined by the formula



where R_1, R_2 and R_3 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl with the proviso that no more than one of R_1, R_2 and R_3 is H; and

and R_4 and R_5 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl.

6. A silica dispersion of Claim 5, wherein at least one of said amino alcohols is 2-amino-2-methyl-1-propanol (AMP).

7. A silica dispersion of Claim 5, wherein at least one of said amino alcohols is 2-amino-2-ethyl-1,3-propanediol (AEPD).

8. A silica dispersion of Claim 5, wherein at least one of said amino alcohols is 2-amino-2-methyl-1,3-propanediol (AMPD).

9. A silica dispersion of Claim 5, wherein at least one of said amino alcohols is 2-amino-1-butanol (AB).

10. A silica dispersion of Claim 5, wherein at least one of said amino alcohols is 2-amino-2-hydroxymethyl-1,3-propanediol (TA).

11. A silica dispersion of Claim 1, further comprising additives selected from biocides, pH modifiers, and dispersants.

12. A method of producing a stable aqueous silica dispersion comprising:

(a) mixing a predetermined effective amount of at least one amino alcohol from the group consisting of primary, secondary, and tertiary amines in which the nitrogen atom of the amine is bonded to a secondary or tertiary carbon atom into a sufficient amount of water;

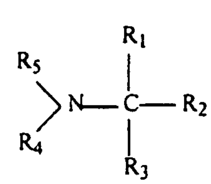
(b) mixing into the amino-alcohol-water mixture of (a) a predetermined amount of silica particles having an average diameter up to about 10 μm or less to produce said stable silica dispersion.

13. The method of Claim 12, wherein said predetermined amount of amino-alcohols is about 1-10 wt % based on the amount of the silica particles of (b).

14. The method of Claim 12, wherein said silica is added in the amount of about 1-30 wt % based on the water used in (a).

15. The method of Claim 12, wherein said silica particles have an average diameter of about 0.01 to 1.5 μm.

16. The method of Claim 12, wherein said amino alcohols are defined by the formula



where R_1, R_2 and R_3 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl with the proviso that no more than one of R_1, R_2 and R_3 is H;

and R_4 and R_5 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl.

17. The method of Claim 16. wherein at least one of said amino alcohols is 2-amino-2-methyl-1-propanol (AMP).

18. The method of Claim 16, wherein at least one of said amino alcohols is 2-amino-2-ethyl-1,3-propanediol (AEPD).

19. The method of Claim 16, wherein at least one of said amino alcohols is 2-amino-2-methyl-1,3-propanediol (AMPD).

20. The method of Claim 16, wherein at least one of said amino alcohols is 2-amino-2-hydroxymethyl-1,3-propanediol (TA).

21. The method of Claim 16, wherein at least one of said amino alcohols is 2-amino-1-butanol (AB).

22. The method of Claim 12, further comprising:

(c) including in steps (a) and (b) additives selected from biocides, pH modifiers, and dispersants.

23. A method of producing an aqueous silica dispersion capable of remaining non-dilatant when subjected to high shear stress comprising:

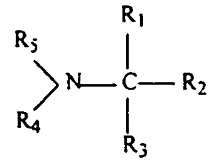
(a) mixing a predetermined effective amount of at least one amino alcohol from the group consisting of primary, secondary, and tertiary amines in which the nitrogen atom of the amine is bonded to a secondary or tertiary carbon atom into a sufficient amount of water;

(b) mixing into the amino-alcohol-water mixture of (a) a predetermined amount of silica particles having an average diameter up to about 10 μm or less to produce said stable silica dispersion.

24. A method of Claim 23, wherein said silica particles have an average diameter of about 0.01 to 1.5 μm.

25. A method of producing an aqueous silica dispersion capable of remaining non-dilatant when subjected to high shear stress comprising:

(a) mixing about 1-10 wt % of at least one amino alcohol based on the silica particles of (b) and having the formula



where R_1, R_2 and R_3 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl with the proviso that no more than one of R_1, R_2 and R_3 is H,

R_4 and R_5 are independently H, C_1-C_{10} alkyl, C_1-C_{10} alkanol, C_1-C_6 cycloalkyl, C_1-C_6 cycloalkanol, aryl, or alkylaryl,

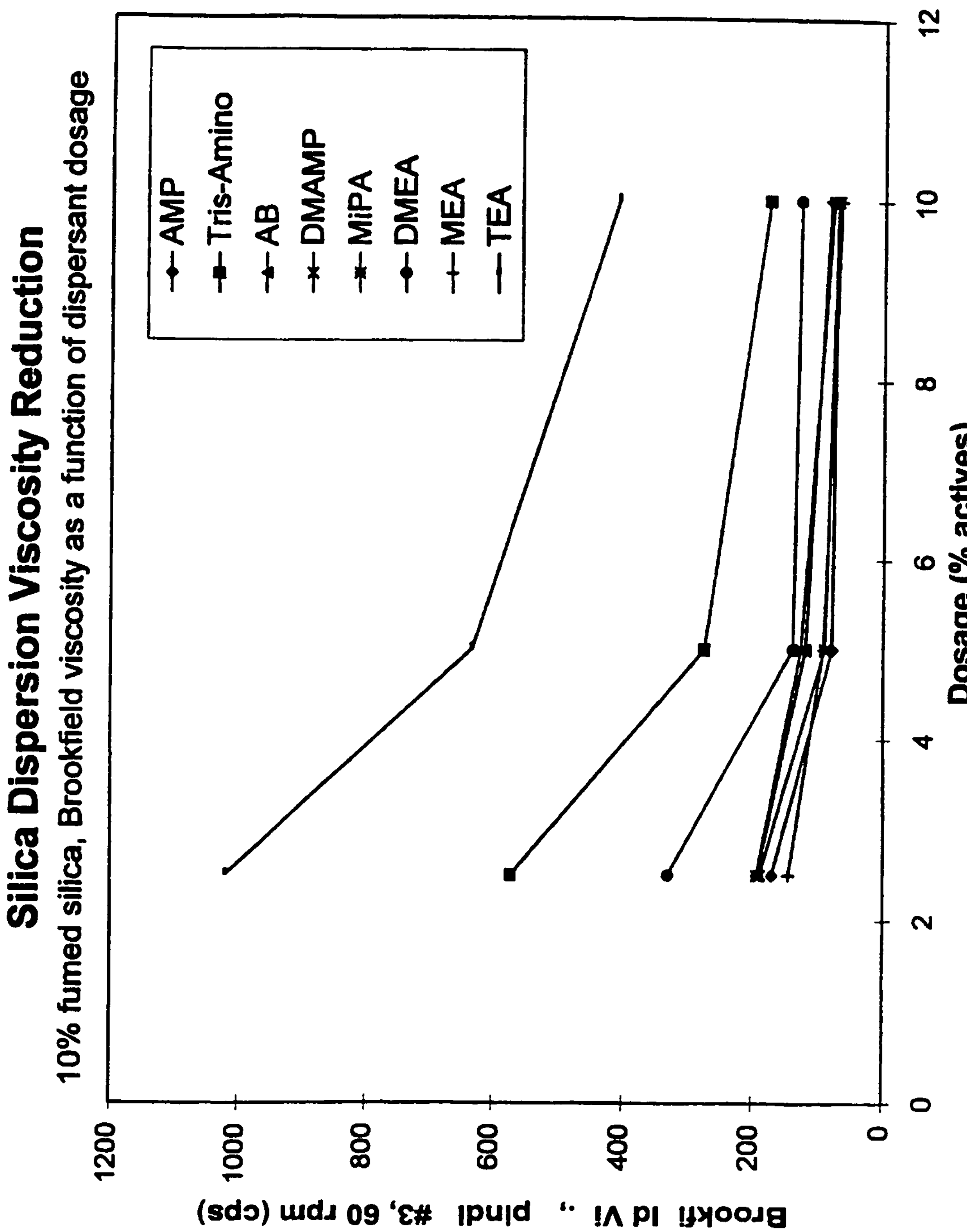
into a sufficient amount of water;

(b) mixing into the amino-alcohol-water mixture of (a) 1-30 wt % of silica particles based on the water of (a), said particles having an average diameter of 1.5 μm or less to produce said stable silica dispersion.

26. The method of Claim 25, wherein said silica dispersion remains non-dilatant at shear stresses above about 10 Pa.

27. The method of Claim 25, further comprising:

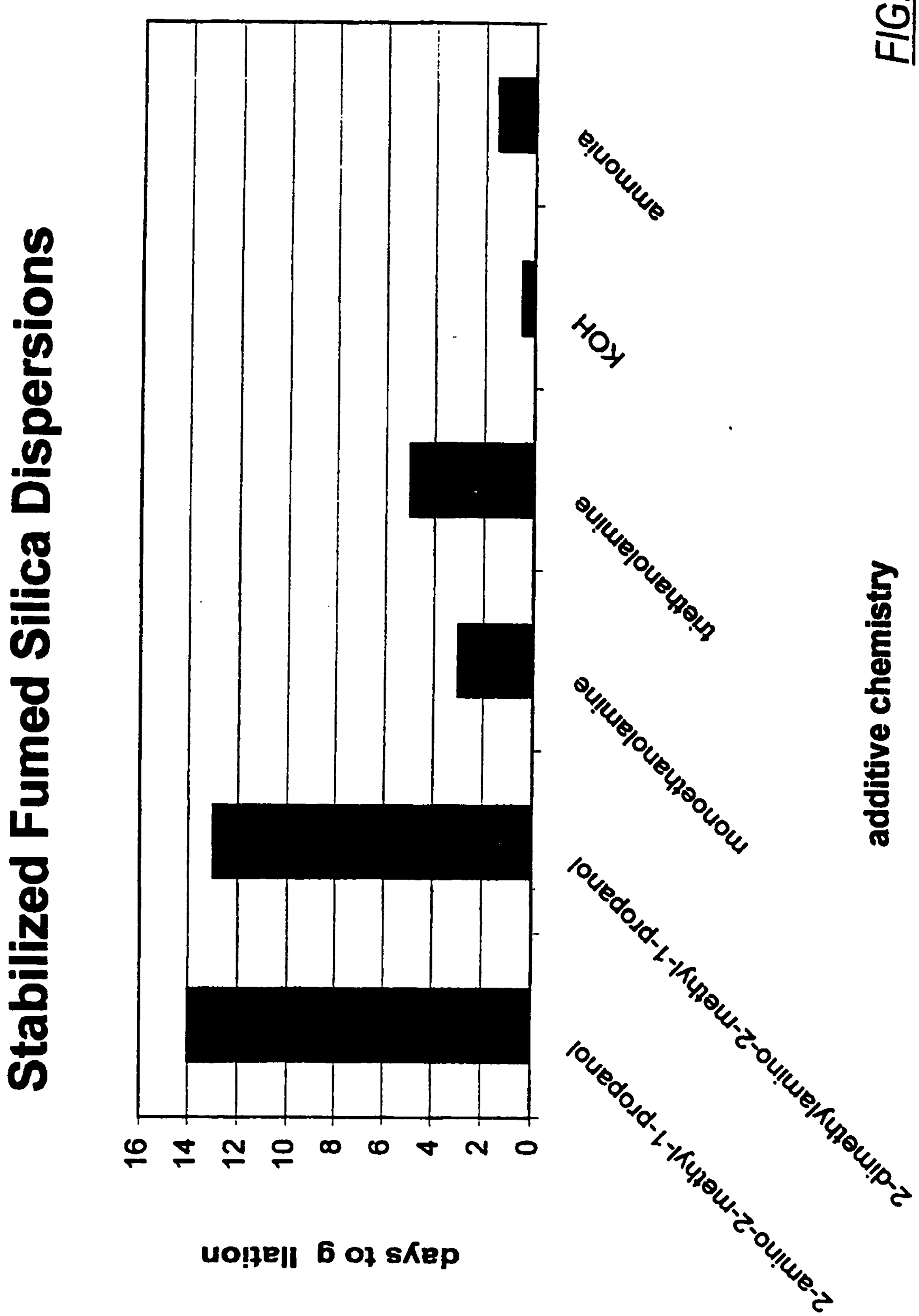
(c) including in steps (a) and (b) additives selected from biocides, pH modifiers, and dispersants.



Silica Dispersion Viscosity Reduction
10% fumed silica, Brookfield viscosity as a function of dispersant dosage
AMP
Tris-Amino
AB
DMAMP
MiPA
DMEA
MEA
TEA
0
2
4
6
8
10
12
Dosage (% actives)
0
200
400
600
800
1000
1200
Brookfi ld Vi ., pindl #3, 60 rpm (cps)

FIG. 1

FIG. 2


Stabilized Fumed Silica Dispersions
days to g llation
16
14
12
10
8
6
4
2
0
ammonia
KOH
triethanolamine
monoethanolamine
2-dimethylamino-2-methyl-1-propanol
2-amino-2-methyl-1-propanol
additive chemistry

Stabilized Fumed Silica Slurry

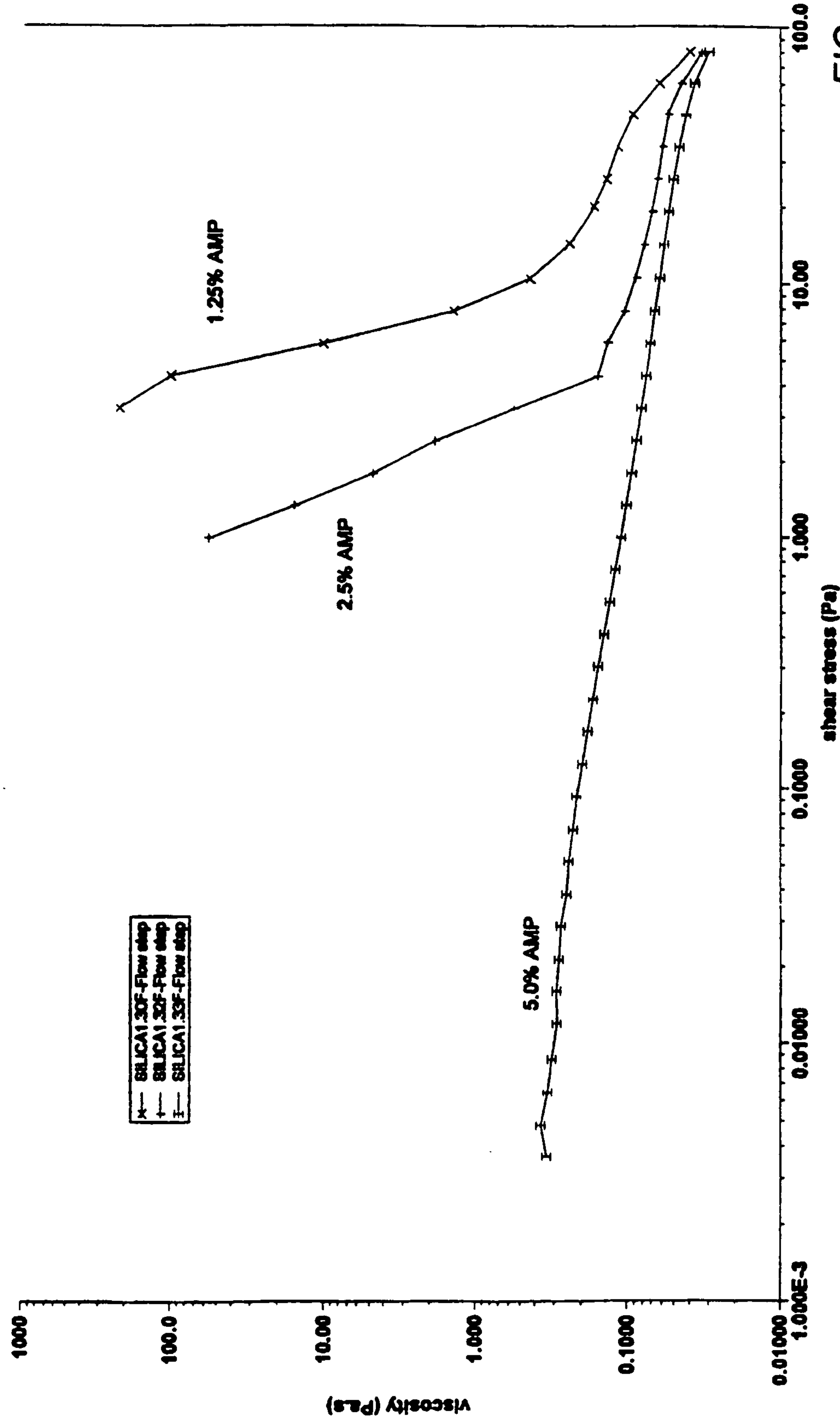


FIG. 3a

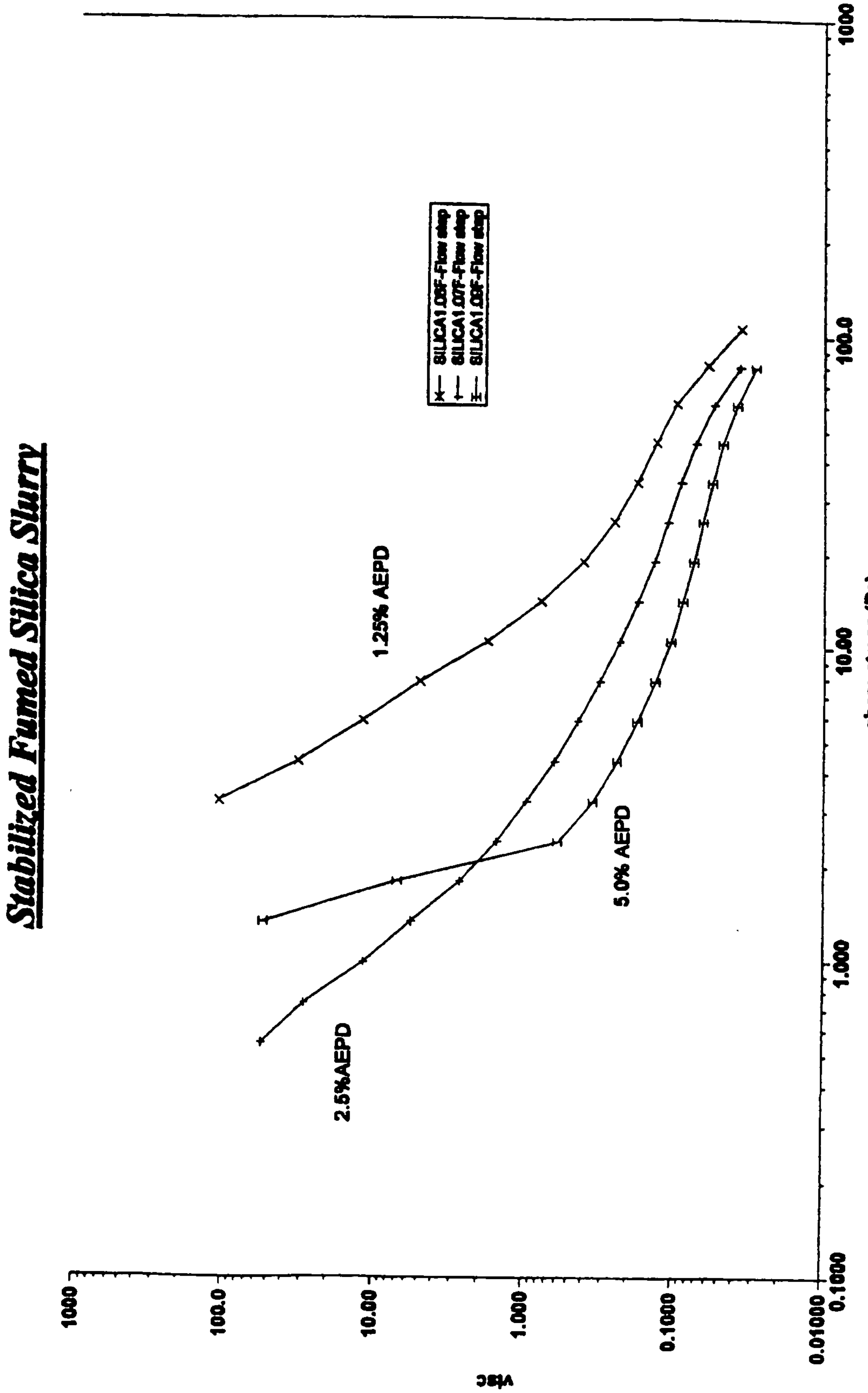

Stabilized Fumed Silica Slurry
SILICA1.08F-Flow step
SILICA1.07F-Flow step
SILICA1.09F-Flow step
1.25% AEPD
2.5%AEPD
5.0% AEPD
visc
1000
100.0
10.00
1.000
0.1000
0.01000
0.1000
1.000
10.00
100.0
1000
shear stress (Pa)

FIG. 3b

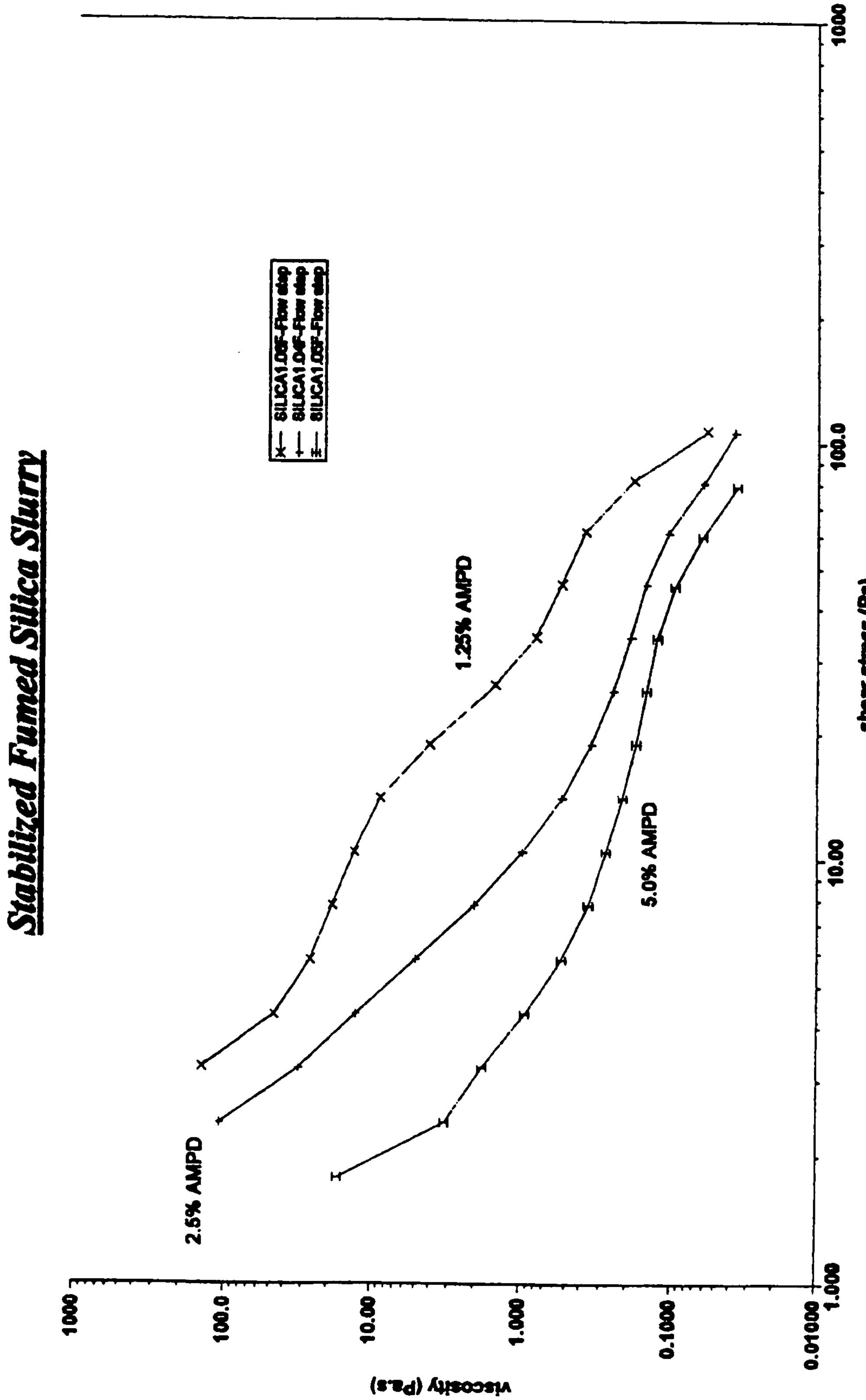

Stabilized Fumed Silica Slurry
1.25% AMPD
2.5% AMPD
5.0% AMPD
shear stress (Pa)
viscosity (Pa.s)
1.000
10.00
100.0
1000
0.01000
0.1000
1.000
10.00
100.0
1000

FIG. 3c

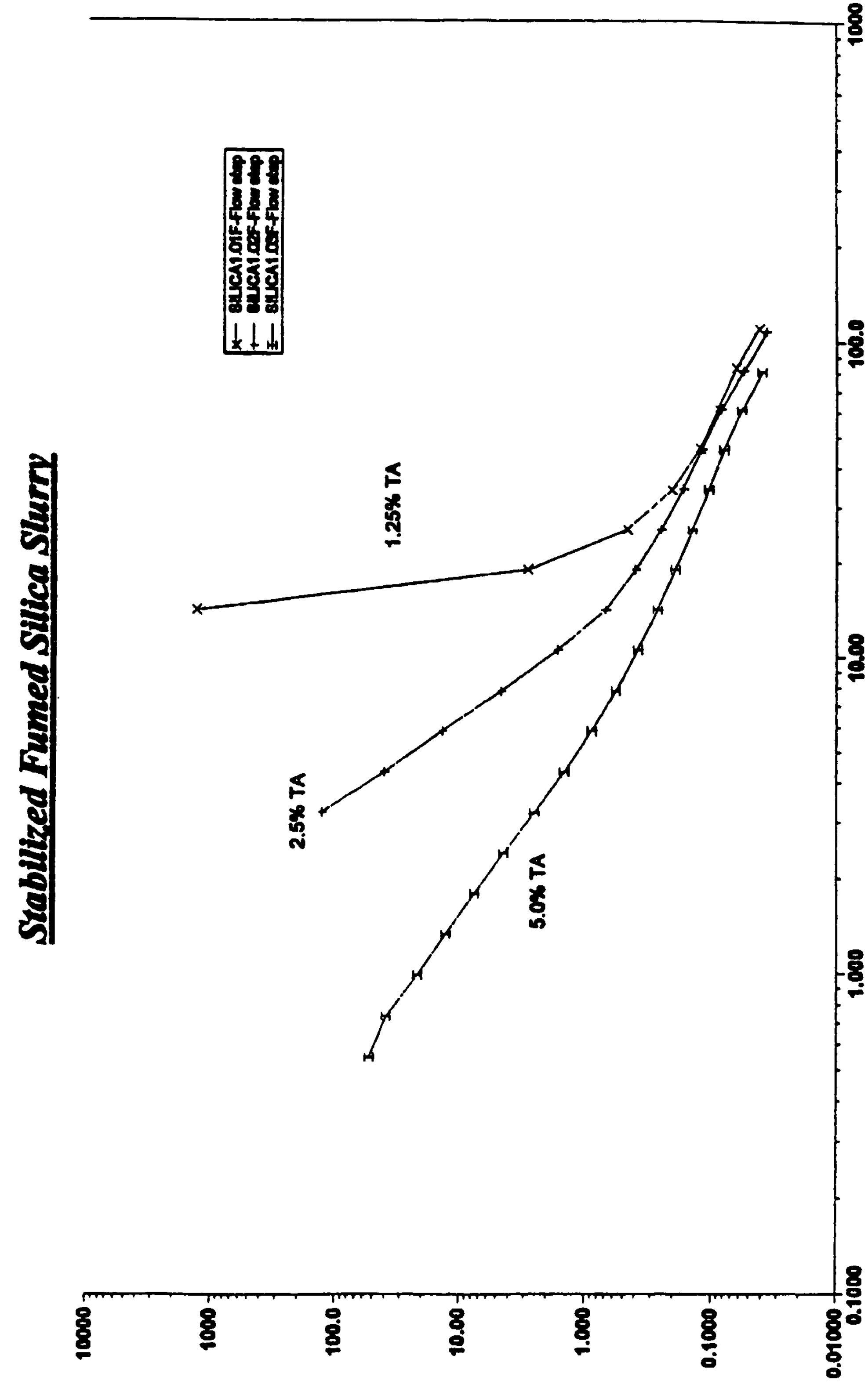

Stabilized Fumed Silica Slurry
SILICA1.01F-Flow step
SILICA1.02F-Flow step
SILICA1.03F-Flow step
1.25% TA
2.5% TA
5.0% TA
viscosity (Pa.s)
10000
1000
100.0
10.00
1.000
0.1000
0.01000
0.1000
1.000
10.00
100.0
1000
shear stress (Pa)

FIG. 3d

Stabilized Fumed Silica Slurry

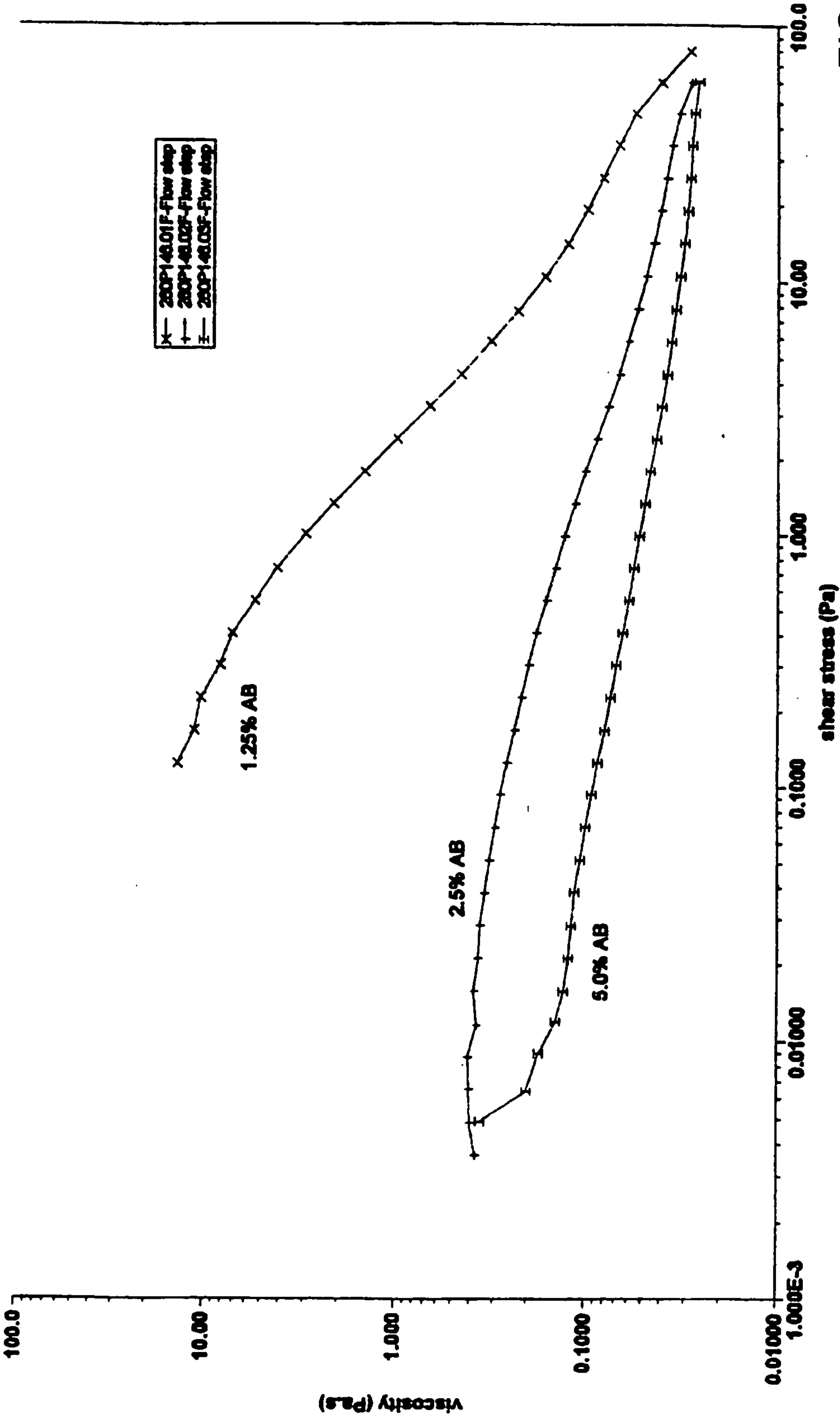


FIG. 3e

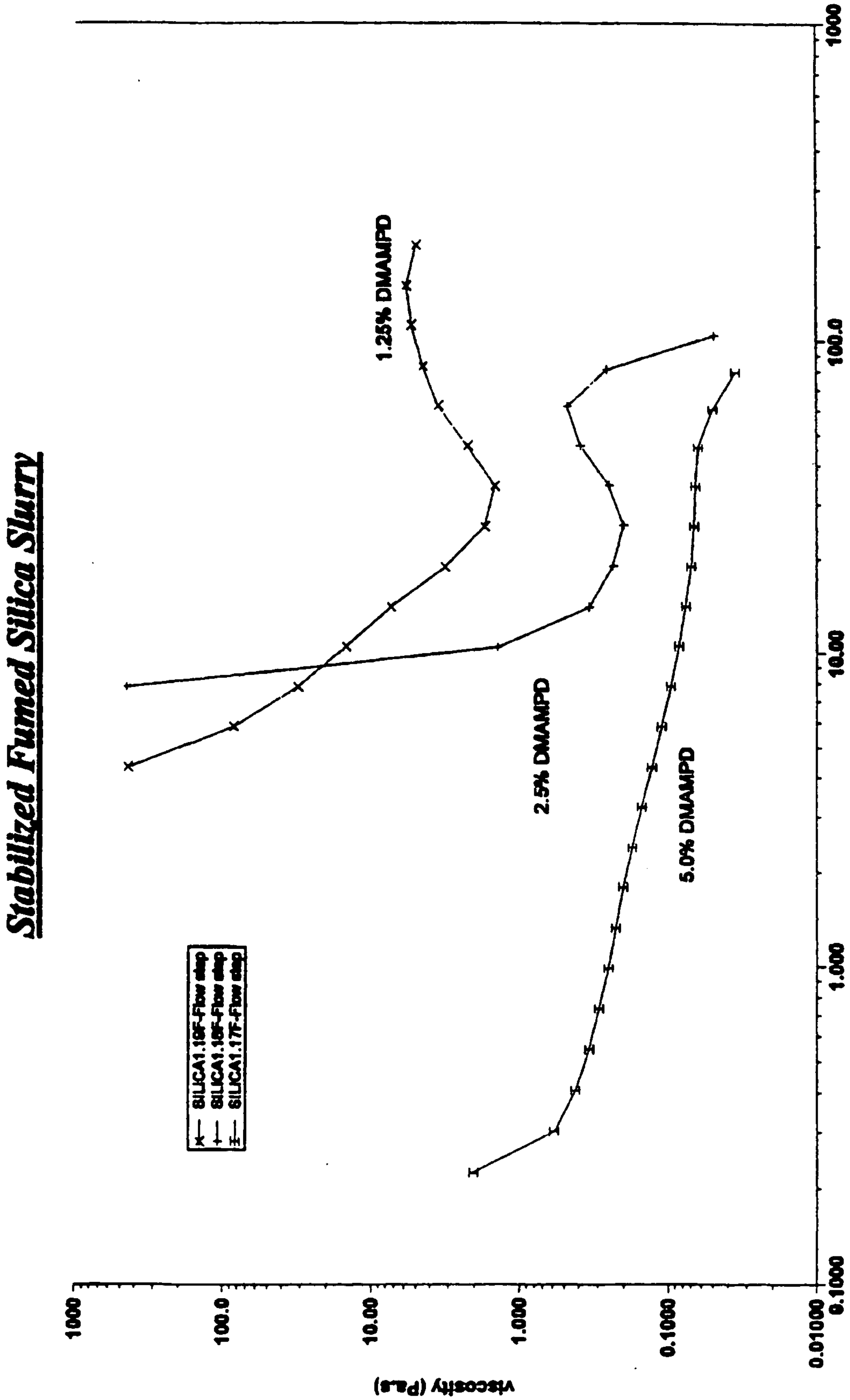

Stabilized Fumed Silica Slurry
SILICA1.19F-Flow step
SILICA1.18F-Flow step
SILICA1.17F-Flow step
1.25% DMAMPD
2.5% DMAMPD
5.0% DMAMPD
viscosity (Pa.s)
shear stress (Pa)
1000
100.0
10.00
1.000
0.1000
0.01000
0.1000
1.000
10.00
100.0
1000

FIG. 3f

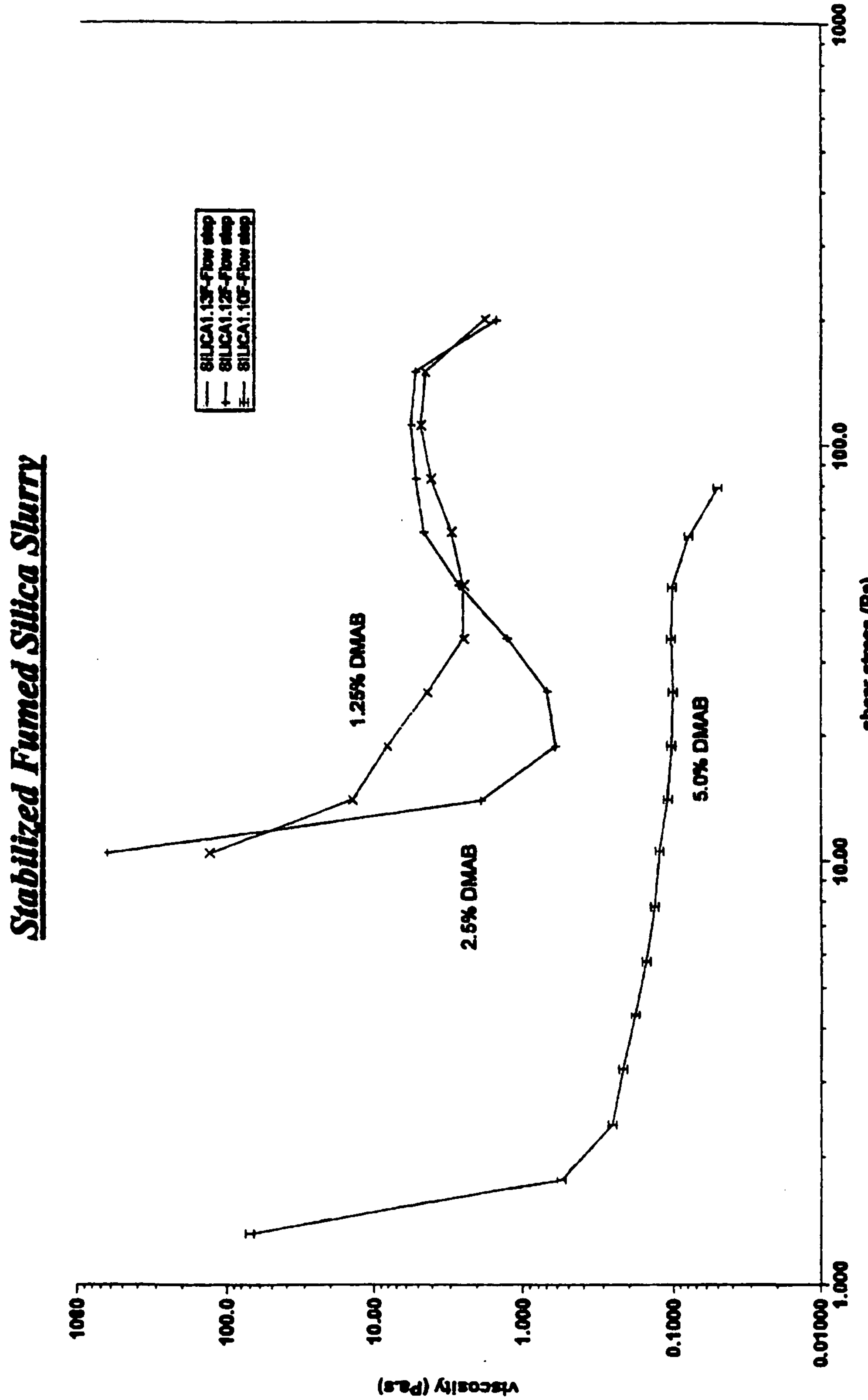

Stabilized Fumed Silica Slurry
SILICA1.13F-Flow step
SILICA1.12F-Flow step
SILICA1.10F-Flow step
1.25% DMAB
2.5% DMAB
5.0% DMAB
viscosity (Pa.s)
shear stress (Pa)
1000
100.0
10.00
1.000
0.1000
0.01000
1.000
10.00
100.0
1000

FIG. 3g

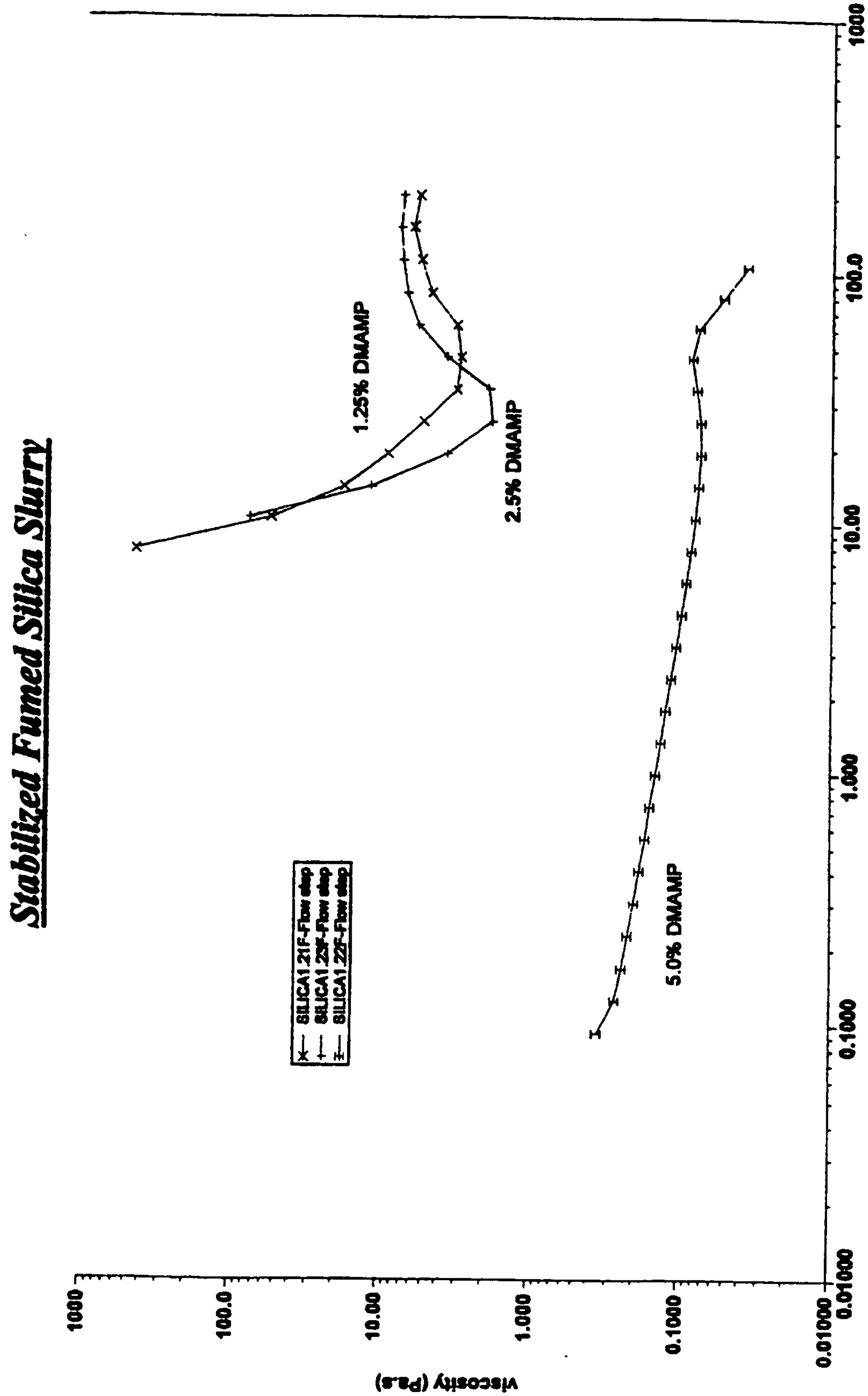

Stabilized Fumed Silica Slurry
SILICA1.21F-Flow step
1.25% DMAMP
2.5% DMAMP
5.0% DMAMP
viscosity (Pa.s)
shear stress (Pa)
1000
100.0
10.00
1.000
0.1000
0.01000

FIG. 3h

Stabilized Fumed Silica Slurry

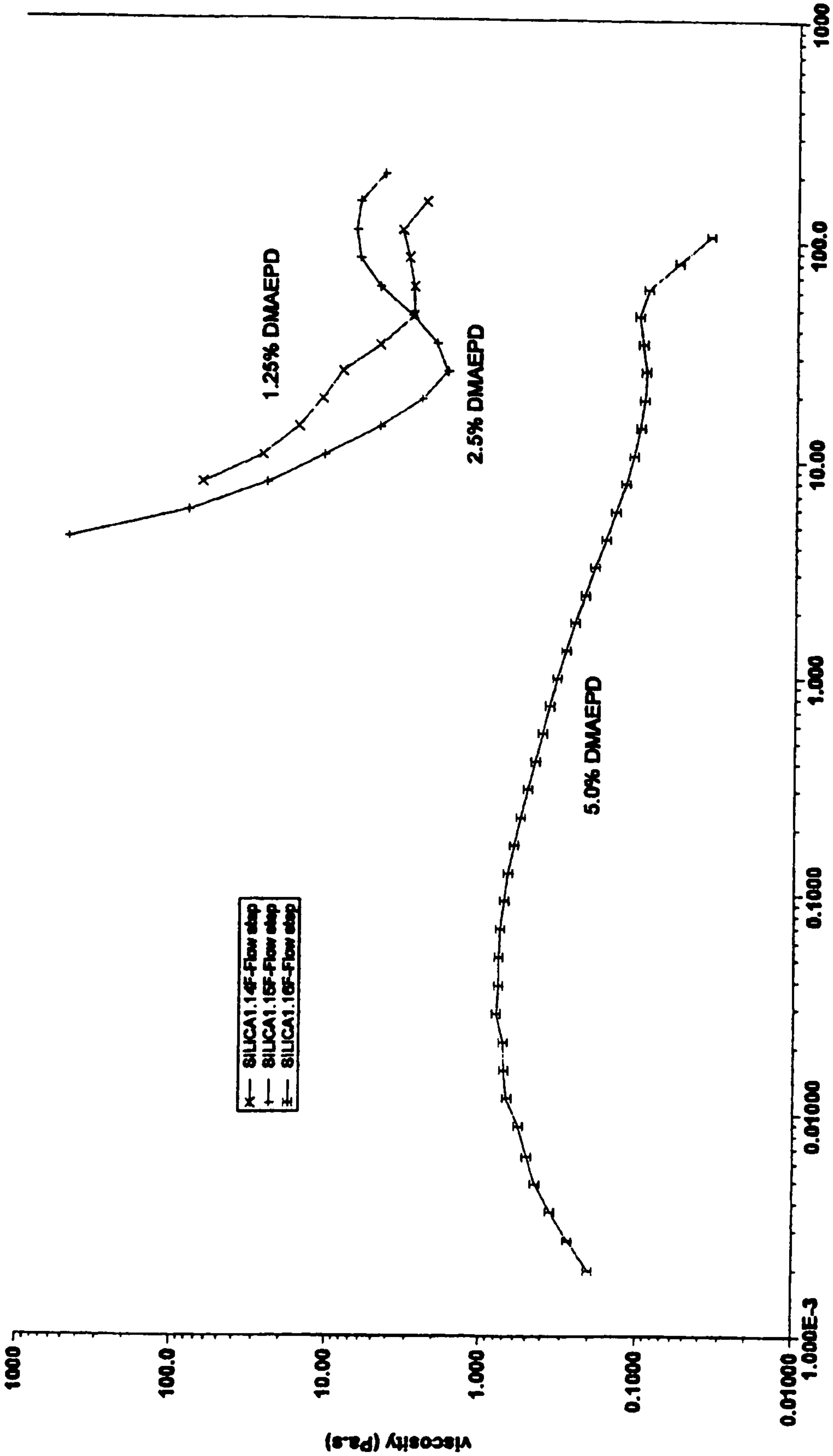


FIG. 3i

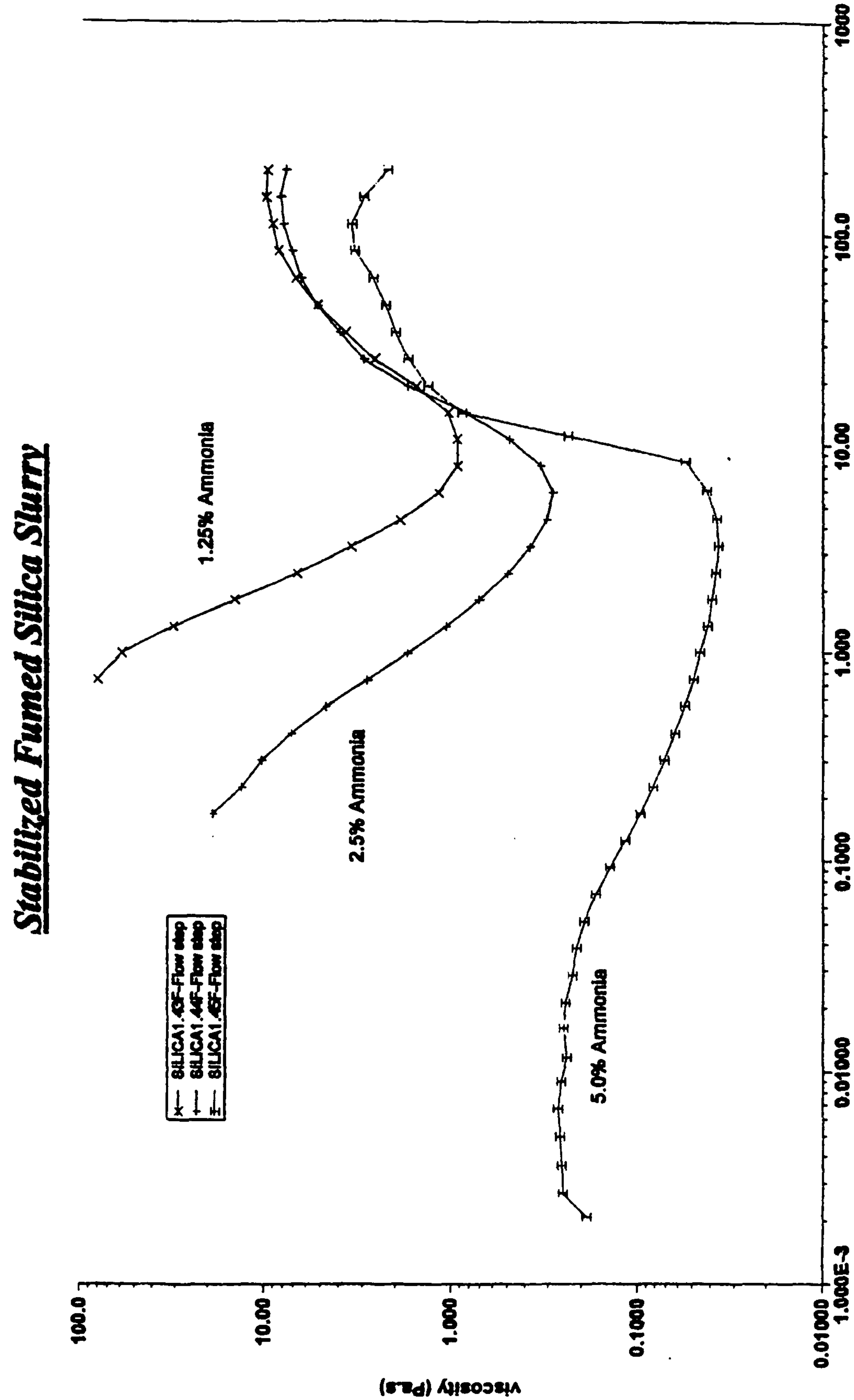

Stabilized Fumed Silica Slurry
SILICA1.44F-Flow step
1.25% Ammonia
2.5% Ammonia
5.0% Ammonia
viscosity (Pa.s)
100.0
10.00
1.000
0.1000
0.01000
shear stress (Pa)
1.000E-3
0.01000
0.1000
1.000
10.00
100.0
1000

FIG. 3j

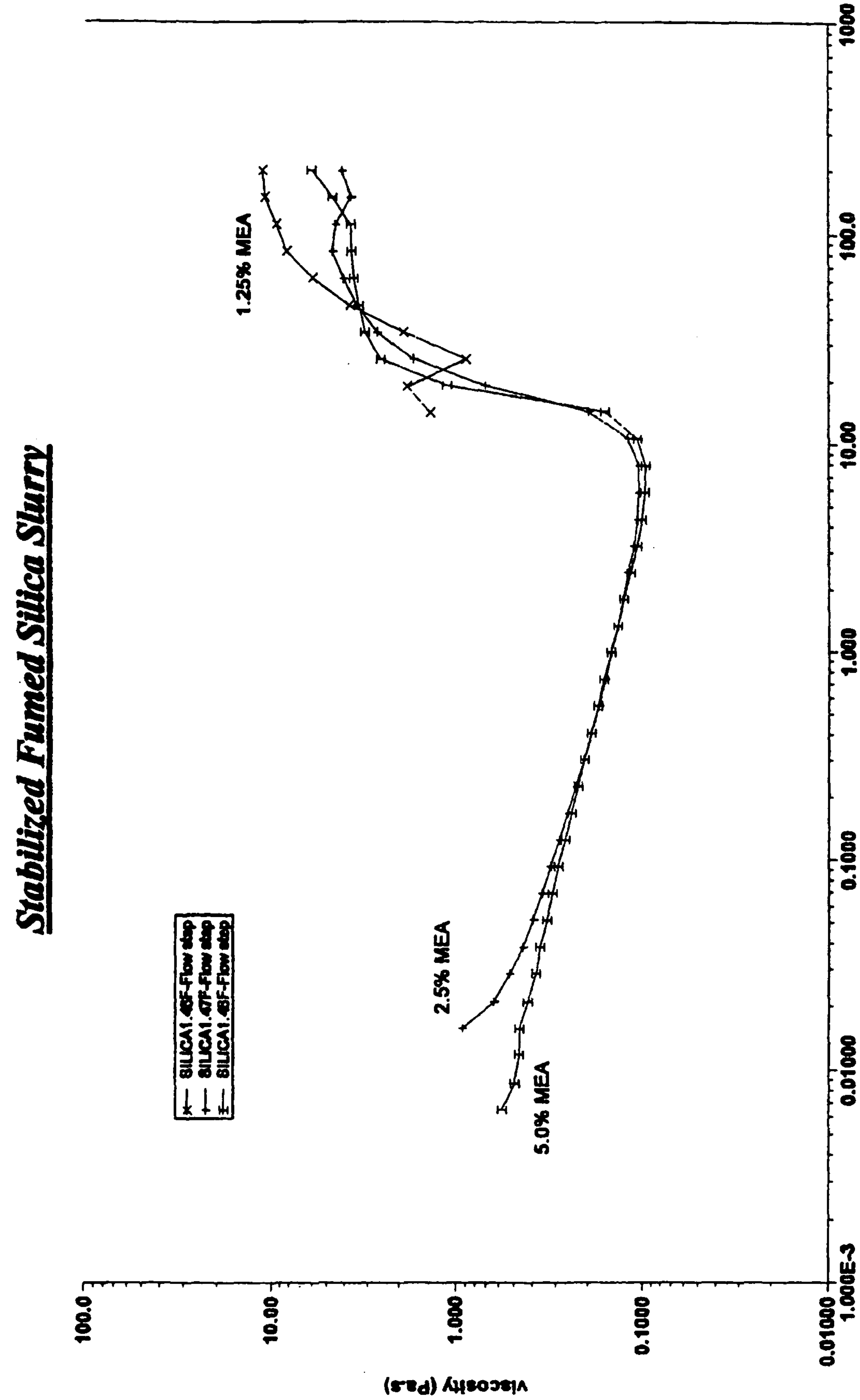

Stabilized Fumed Silica Slurry
viscosity (Pa-s)
100.0
10.00
1.000
0.1000
0.01000
shear stress (Pa)
1.000E-3
0.01000
0.1000
1.000
10.00
100.0
1000
1.25% MEA
2.5% MEA
5.0% MEA

FIG. 3k

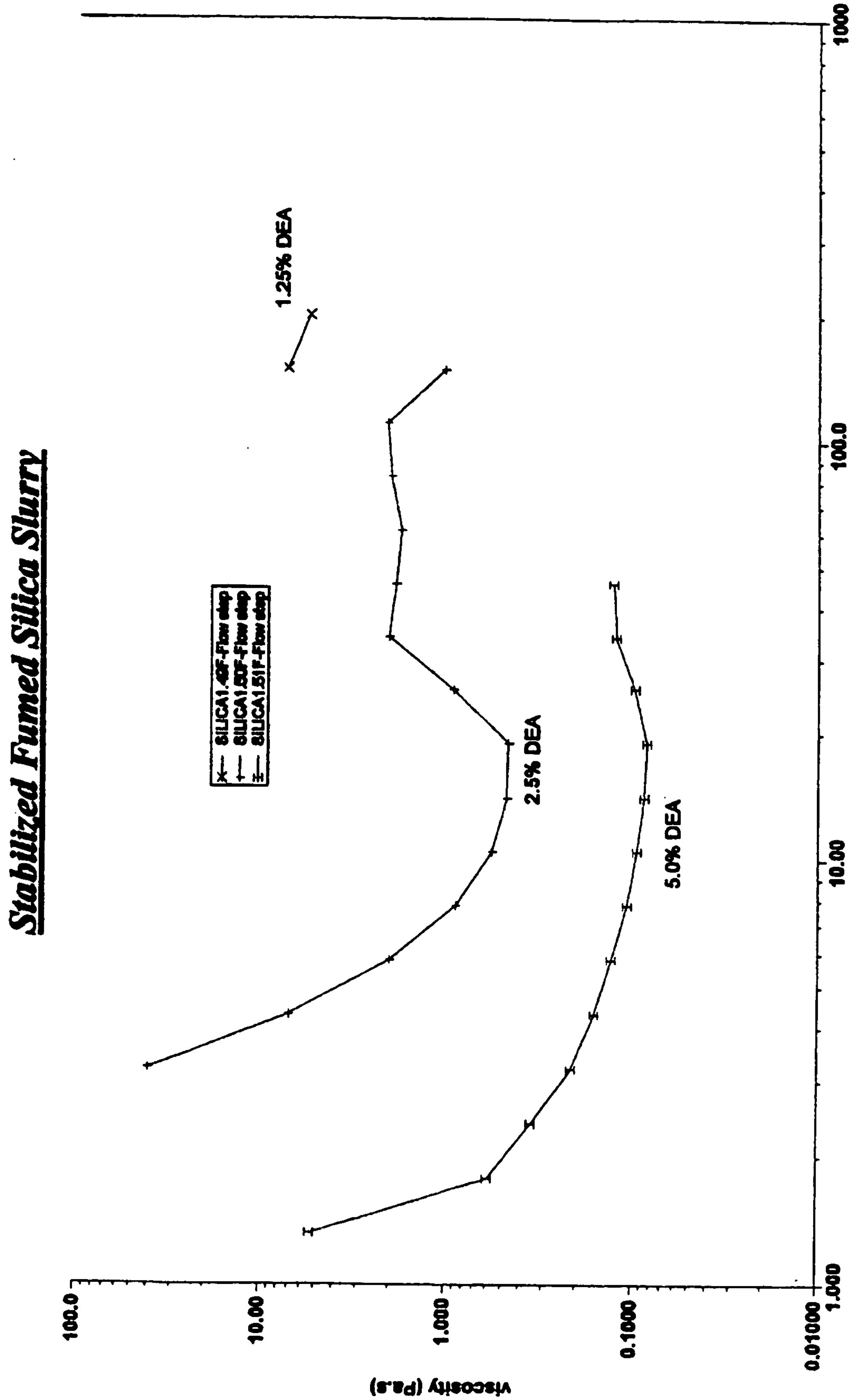

Stabilized Fumed Silica Slurry
1.25% DEA
2.5% DEA
5.0% DEA
shear stress (Pa)
viscosity (Pa.s)
1.000
10.00
100.0
1000
0.01000
0.1000
1.000
10.00
100.0

FIG. 3I

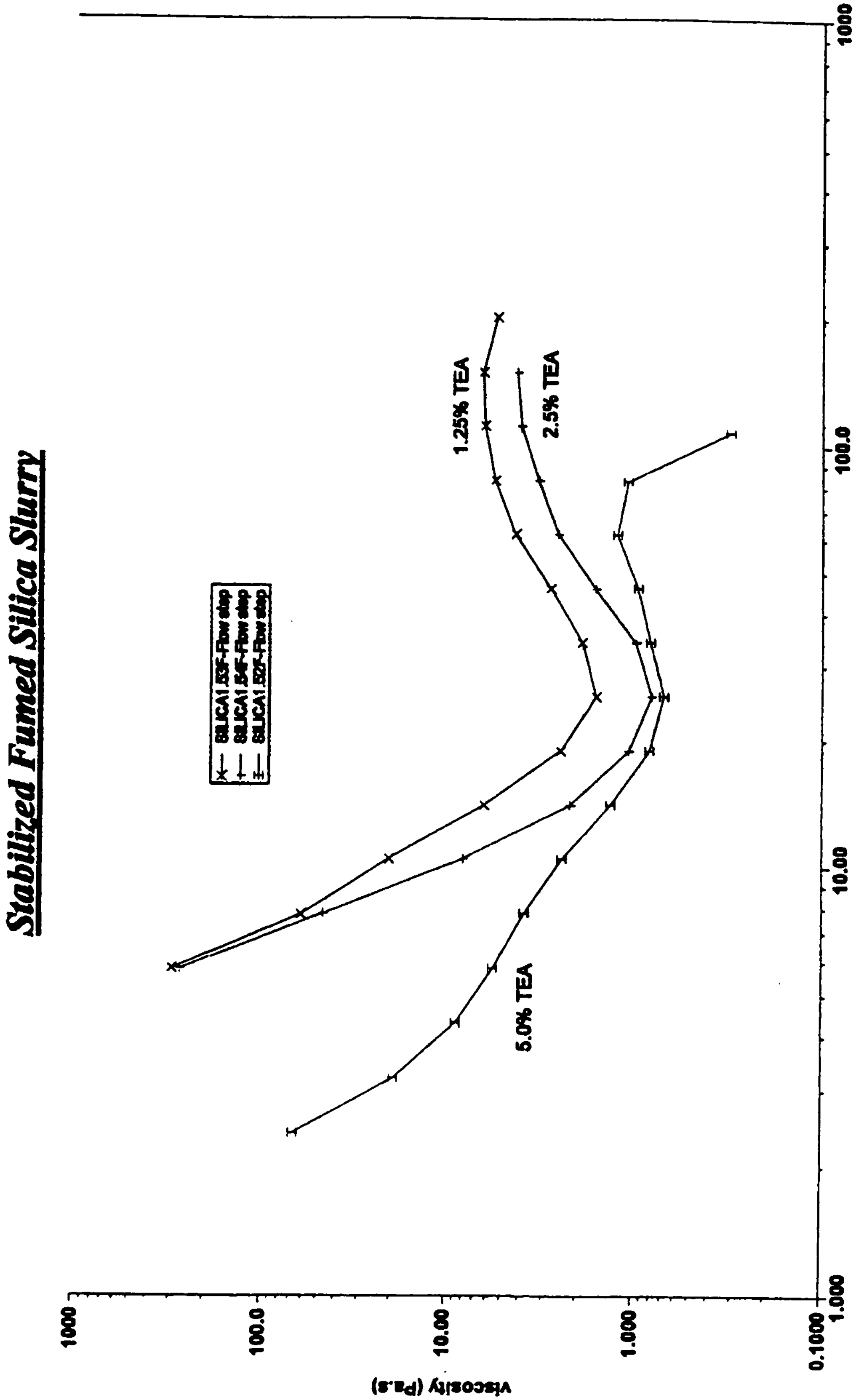

Stabilized Fumed Silica Slurry
SILICA1.53F-Flow step
SILICA1.54F-Flow step
SILICA1.52F-Flow step
1.25% TEA
2.5% TEA
5.0% TEA
viscosity (Pa.s)
1000
100.0
10.00
1.000
0.1000
1.000
10.00
100.0
1000
shear stress (Pa)

FIG. 3m

Stabilized Fumed Silica Slurry

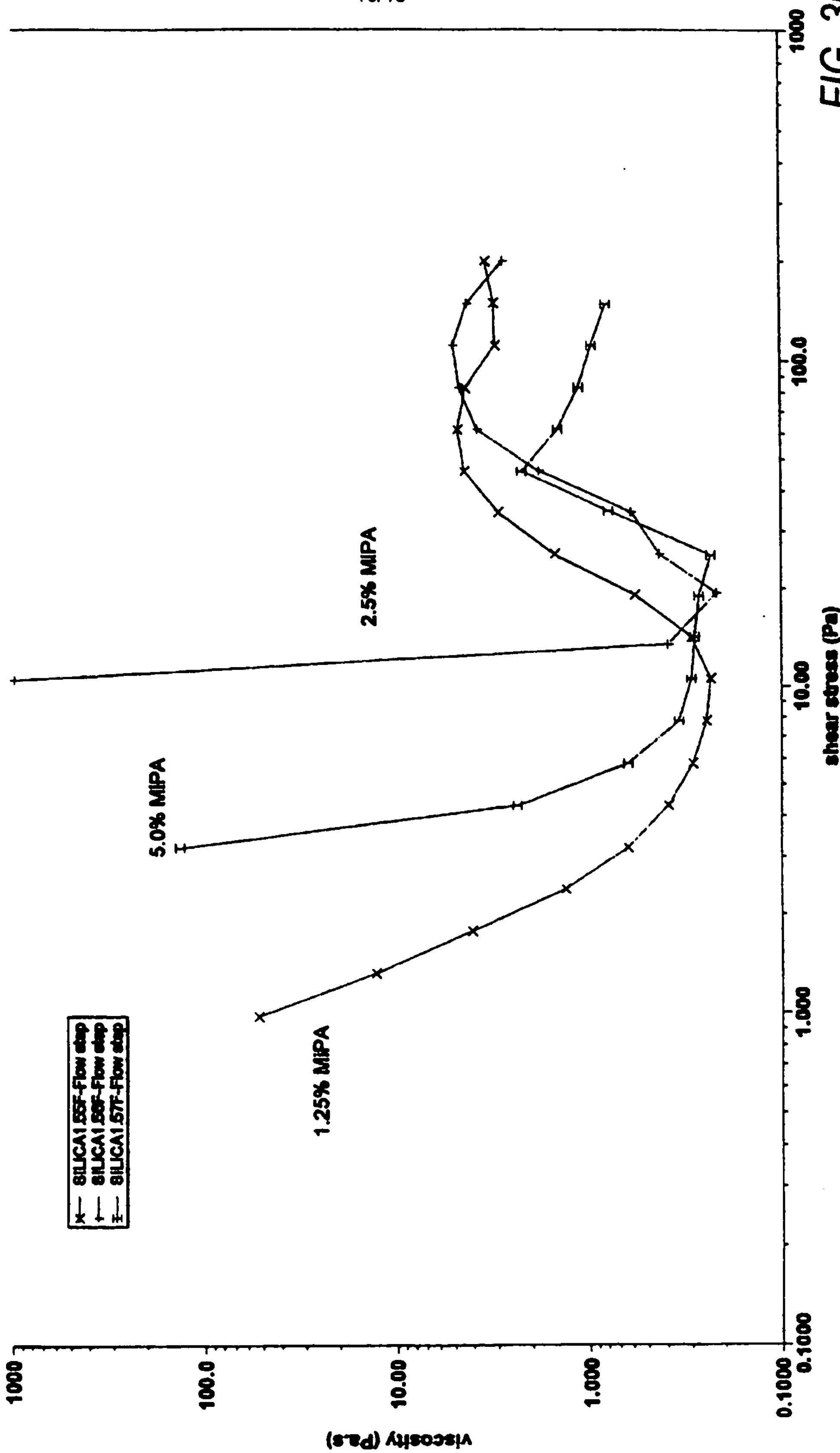


FIG. 3n

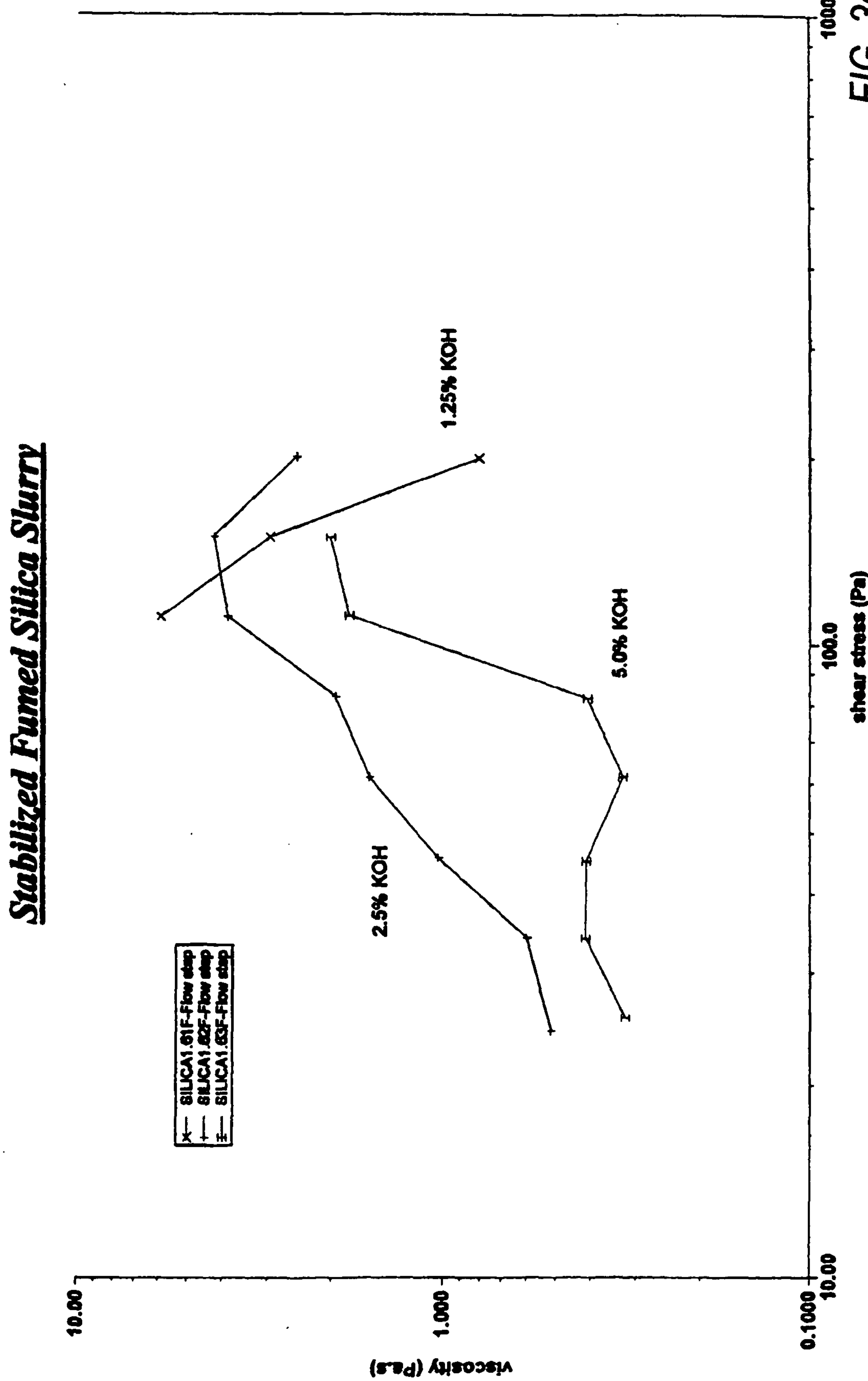

Stabilized Fumed Silica Slurry
SILICA1.61F-Flow step
SILICA1.62F-Flow step
SILICA1.63F-Flow step
1.25% KOH
2.5% KOH
5.0% KOH
viscosity (Pa.s)
10.00
1.000
0.1000
shear stress (Pa)
10.00
100.0
1000

FIG. 30

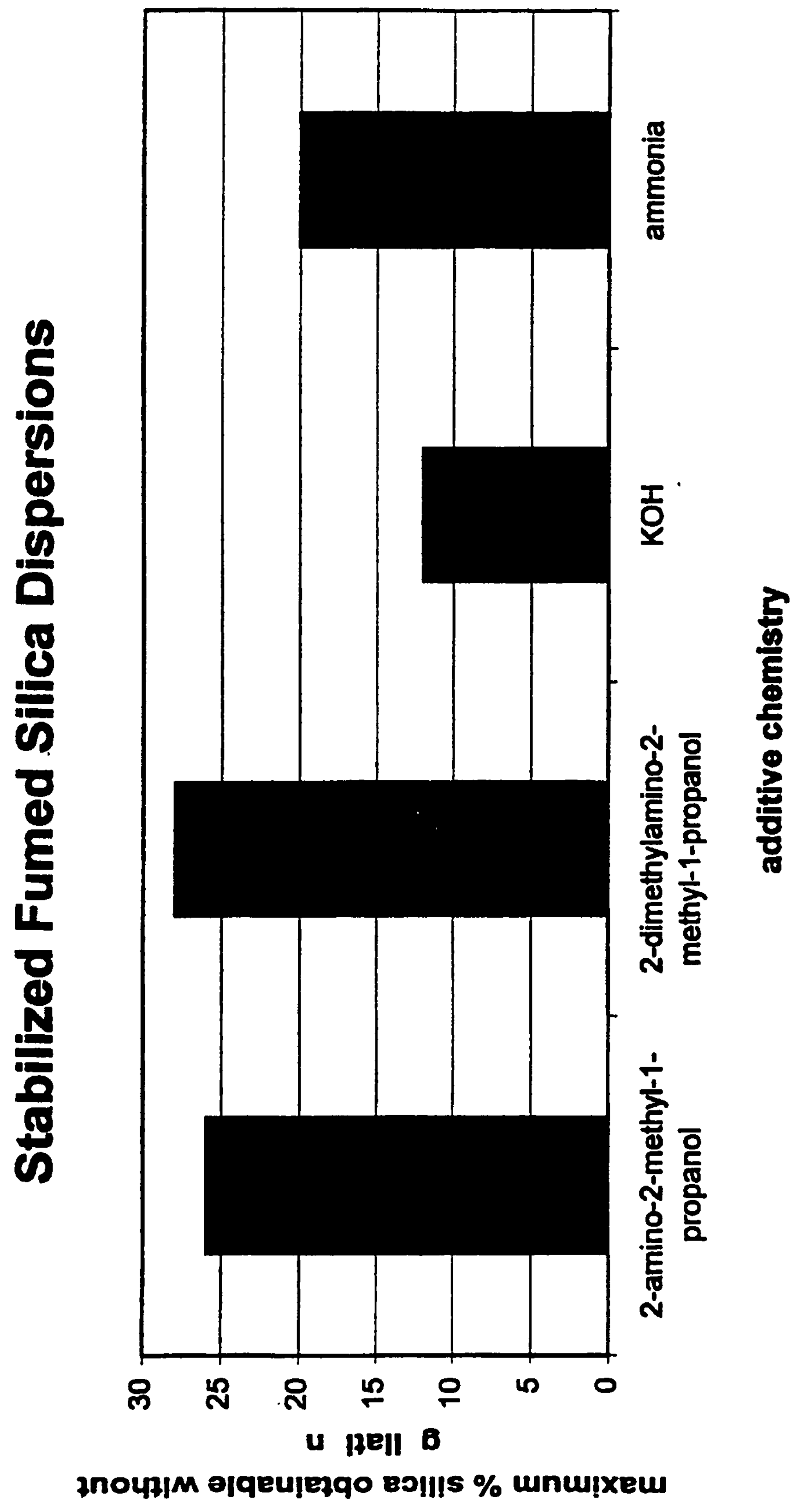

Stabilized Fumed Silica Dispersions
maximum % silica obtainable without
30
25
20
15
10
5
0
2-amino-2-methyl-1-propanol
2-dimethylamino-2-methyl-1-propanol
KOH
ammonia
additive chemistry

FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/11849

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :BO1J 13/00; CO1B 33/148
US CL :516/81, 86, 87; 106/287.34
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : BO1J 13/00; CO1B 33/148; 516/81, 86, 87; 106/287.34

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,176,752 A (SCHEINER) 05 January 1993 abstract; column 3, line 13 - column 4, line 46; and Examples 1 and 2.	1-27
A	US, 2,601,352 A (WOLTER) 24 June 1952 column 6, line 23 - column 7, line 32.	1-27
A	US, 2,799,658 A (NICKERSON) 16 July 1957 Example II; and column 4, lines 31-43.	1-27
A	US, 2,984,629 A (LOFTMAN ET AL.) 16 May 1961 column 2, line 43 - column 3, line 5.	1-27
A	US, 3,630,954 A (YATES) 28 December 1971 column 1, line 68 - column 3, line 60.	1-27

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
09 AUGUST 1999	14 SEP 1999

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Authorized officer
RICHARD D. LOVERING
Telephone No. (703) 308-0443

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/11849

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, 5,221,497 A (WATANABE ET AL.) 22 June 1993 abstract; column 7, line 40 - column 8, line 29; and Example 5.	1-27